

Remarks

Applicants have received and reviewed the Office Action dated October 14, 2008. By way of response, Applicants have cancelled claim 5 without prejudice. Applicants have amended claims 1, 11, 13 and 17-21. Applicants have added new claims 22 and 23. Claims 1-4 and 6-23 are pending. Applicants assert that no new matter has been added.

35 U.S.C. § 112

Claims 1-21 were rejected under 35 U.S.C. 112, second paragraph, as being indefinite. Applicants respectfully traverse this rejection.

Applicants have amended claims 1, 11, 13 and 17-21 to particularly point out and distinctly claim the subject matter which Applicants regard as the invention. Applicants believe the claims comply with 35 U.S.C. 112, second paragraph, and respectfully request withdrawal of this rejection.

35 U.S.C. § 102(b) and 103(a)

Claims 1-21 were rejected under 35 U.S.C 102(b) as anticipated by Marihart, U.S. 4,786,307 or in the alternative as being obvious over Marihart '307 under 35 U.S.C. § 103(a). Claims 17-21 were rejected under 35 U.S.C 102(b) as anticipated by Marihart, US 4,698,090 or in the alternative, as being obvious over Marihart '090 under 35 U.S.C. § 103(a). Claims 1-16 were rejected as being obvious over Marihart '090 under 35 U.S.C. § 103(a). Applicants respectfully traverse these rejections.

Solely to further prosecution and without acquiescing to the rejections, Applicants have amended claim 1 to further clarify the invention. Claim 1 now recites a process for preparing a fertilizer composition, comprising the following steps: reacting a humified fossil material, which is selected from the group consisting of leonardite, lignite, xylite and peat, in water in the presence of gluconic acid, wherein the gluconic acid is added to said fossil material in such a quantity so as to adjust the pH of the reaction medium to a value of less than 2.5; and subsequently adding to the mixture an alkaline agent in such a quantity as to adjust the pH to a basic value.

Neither the '307 nor the '090 reference disclose a process for preparing a fertilizer composition wherein the gluconic acid is added to said fossil material in such a quantity so as to adjust the pH of the reaction medium to a value of less than 2.5. In fact, the '307 patent specifically recites a pH above 2.5, usually in the range from about 4 to 6 ('307 Col 2, lines 10 & 58-64). The '090 patent does not disclose or suggest the use of gluconic acid. Instead, the '090 patent discloses the use of sodium gluconate, a sodium salt of gluconic acid ('090 Col 4, lines 40-43).

Neither the '307 nor the '090 reference disclose each and every limitation of the presently claimed invention. Applicants respectfully request withdrawal of the 102(b) rejections.

Applicants submit the presently claimed invention is new, novel, non-obvious and inventive over the cited prior art for at least the following reasons: 1) The use of gluconic acid at a pH below 2.5, instead of a salt of gluconic acid, results in the addition of protons to the leonardite humic material; 2) This addition of protons forces changes in the supramolecular conformation of the humic substances; 3) These changes result in the unexpected extraction of molecules or molecular domains that otherwise could not be extracted by using salts of gluconic acid or gluconic acid at a higher pH.

'090 Reference

The presently claimed invention recites using gluconic acid for the extraction of leonardite humic material. The use of gluconic acid implies addition of protons to the leonardite humic material. By contrast, the '090 reference discloses the use of salts of gluconic acid, which do not include the addition of protons.

Humic substances are supramolecular materials in which a large number of relatively small and heterogeneous molecules are self-associated into aggregates held together, in the flocculated state, by mainly dispersive weak forces (van der Waals, π - π), hydrogen bonds, and electrostatic complexes stabilised by metal bridges.

The multiple mutual relationships between the humic associations are then altered when new and energetically stronger hydrogen bonds are formed by the introduction of protons into the supramolecular system.

The addition of hydroxy acids to humic materials change the associations of humic molecules because of the protonation and proton exchange at the carboxyl sites, thereby altering the stability of the weaker dispersive forces and forcing changes in the supramolecular conformation. The altered humic conformations are then prone to expose inner molecular domains that are available for extraction if the solution pH is sequentially raised to alkaline values.

This mechanism does not happen when only salts were added to the humic material. In the lack of proton additions (gluconic salts do not bring protons), the supramolecular associations are not altered, thereby failing to expose further inner molecules to the subsequent alkaline extraction.

This brief mechanistic description helps to understand why only the use of gluconic acid, instead of sodium gluconates, involves an essential and profound difference in the extraction of humic matter from a source such as a leonardite ore.

The protons supplied to the humic ore by adding the hydroxy acid in the presently claimed invention provides an alteration of the weak molecular arrangement such that the subsequent pH raise with anhydrous ammonia or potassium hydroxide, is capable of extracting molecules or molecular domains that would not be extractable if only the sodium salt of gluconic acid was added to the leonardite ore.

The novel use of gluconic acid instead of a salt thereof unexpectedly results in a superior extraction efficiency compared to the salt extraction disclosed in the '090 reference. The use of gluconic acid for extraction is not disclosed, suggested or even contemplated by the '090 reference.

'307 Reference

An important distinction between the '307 reference and the composition and method of the present application is that the '307 reference discloses an extraction method that employs a first treatment with a hydroxy acid (including gluconic acid) followed by a second treatment with salts of the hydroxy acid. Importantly, the '307 reference discloses an extraction mixture with a pH of "... above about 2.5, usually in the range from 4 to 6 ..." (Col. 2, lines 58-64). By contrast, the present composition utilizes a reaction mixture with pH below 2.5.

Importantly, from the discussion above it is apparent that a conformational rearrangement of the humic ore does not occur if the hydroxy acid is not in the protonated form. This is the case when the extraction with the hydroxy acid is usually performed at pH between 4 and 6 as disclosed and preferred by the '307 reference. Because the pKa of gluconic acid 3.7, only the dissociated form is predominant at these pHs and the alteration of the supramolecular humic structure is not produced due to lack of the necessary protons.

Moreover, had any structural alteration of the leonardite matter occurred, its effect would be reverted or made useless when the salts of the gluconic acid is further added before the final raise to alkaline pHs with ammonia or alkaline hydroxides.

In fact, the subsequent addition of the salts of gluconic acid in the process of the '307 reference increases the volume of the extracting solution, likely resulting in an even further reduction of the protonation (if any) of the previously added hydroxy acid. By contrast, the presently claimed invention utilizes gluconic acid at a pH below 2.5.

The pH used by the '307 reference is higher than the pKa of the acid, thus the predominant form of the acid is already the dissociated one. It is therefore understandable that the addition of the gluconate salt after that of the hydroxy acid does not change the effect on the structure of the leonardite ore since the hydroxy acid was already predominantly dissociated.

The following rise in pH, to conclude the extraction, is ineffective in solubilising the inner biologically active molecules of the humic ore. Hence, the molecular yield obtained by the method of the '307 reference is likely similar to the yield obtained from the '090 reference.

By contrast, the presently claimed invention is based on an extraction with gluconic acid "at a pH below 2.5." The presently claimed invention provides the humic ore with sufficient protons to alter the aggregation state of leonardite and effectively induce an novel and unexpected increased solubilisation of biologically active humic molecules.

Moreover, the '307 reference discloses that after the addition of a hydroxy acid, followed by addition of salts of the hydroxy acid, one or more metal salts (divalent and trivalent metals) are then added to the mixture (Col. 4, lines 1-15) prior to the final rise to an alkaline pH with ammonia or alkaline hydroxide.

This sequence of additions is bound to create the conditions by which, whatever previously solubilised humic material, may form strong complexes with the added divalent and trivalent metals and cause the precipitation of the formed metal-complexes, thereby subtracting humic molecules from the solutions. It is known that divalent and trivalent metals generally form complexes of high stability constants with humic substances at a pH between 4 and 6.

Further, polyvalent cations may function as electrostatic bridges among two or more humic molecules, thereby exceeding their solubilisation potential in water and causing a flocculation of the aggregate composed of multiple metal-complexes. A further rise of the pH does not generally displace the metals from the humic complexes.

This concept is embodied in the extraction procedure recommended by the International Humic Substances Society, whereby the source, from which humic matter must be extracted, is preliminarily treated with dilute mineral acid in order to displace as many salts and metals as possible from the humic acidic functional groups and concomitantly protonise the same functions. Without such acidic treatment, the extraction in alkaline pH would fail to quantitatively extract humic substances.

By contrast, the present invention adds metal salts after treatment with ammonia or alkaline hydroxides. This maximizes the effect of maintaining the metals in solution as salts of their added counter-ions or as their hydroxides, and prevents the loss from solution of humic molecules by precipitation as metal-humic complexes.

Therefore, the presently claimed invention is unexpectedly superior to the '307 reference because the extraction is conducted at a low pH and the addition of metals is conducted at higher pH values, thereby avoiding the re-precipitation of the extracted humic molecules as metal-humic complexes.

Supplemental Experiments Supporting Unexpected Results

A comparison of the presently claimed extraction method and the '090 and '307 methods are below. The selected samples are the following:

1. A granular humic extract obtained by following the presently claimed invention.
2. A commercial liquid product by ACTAGRO obtained by the procedure outlined in the '090 reference that claims to contain 7% of organic acids.

3. A commercial liquid product by ACTAGRO obtained by the procedure outlined in the '307 reference that claims to contain 22% of organic acids.

The liquid commercial ACTAGRO products were freeze-dried in order to obtain a dry product for further analyses. The dry granular extract obtained by a process of the present application was used for analyses without any further manipulation.

Elemental analyses

The elemental content of carbon and nitrogen in the selected samples was obtained in duplicate by employing an Interscience EA1108 elemental analyzer.

Table 1. Elemental C and N content of humic extracts

Element	Sample 1 – Present		Sample 2 – '090		Sample 3 – '307	
	Invention		Reference		Reference	
C	38.3	35.3	6.77	7.89	29.9	30.8
N	0.80	0.87	11.9	12.6	1.09	1.1

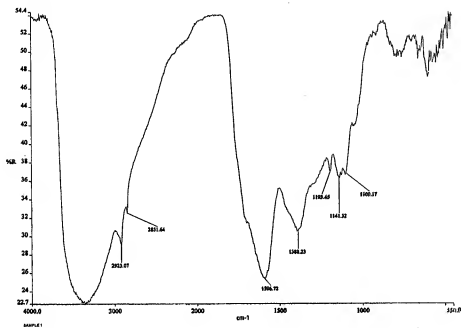
A process of the present invention (Sample 1) has a C content that is significantly larger than Sample 2 (ACTAGRO 7%) and Sample 3 (ACTAGRO 22%). The N content was not substantially different among the samples as one would have expected since no addition of N was claimed in the extraction procedure.

DRIFT, Diffuse Reflectance Infrared Fourier Transform Spectrometry

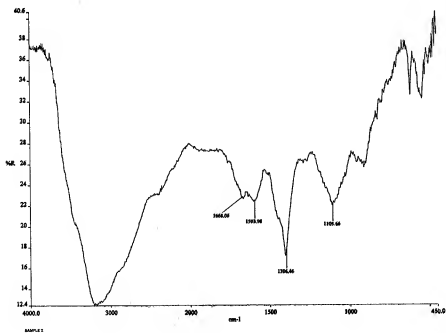
Diffuse Reflectance Infrared Fourier Transform (DRIFT) spectra of humic samples were recorded with a Perkin Elmer 1720-X FT-IR spectrometer, equipped with a Perkin-Elmer Diffuse Reflectance accessory, accumulating up to 100 scans with a resolution of 4 cm⁻¹. Before DRIFT analysis, the solid and freeze-dried samples were first thoroughly mixed to obtain a representative sample and then finely ground in a swinging mill and diluted with KBr powder (2.5/100, w/w) in an agate mortar.

The following spectra were obtained:

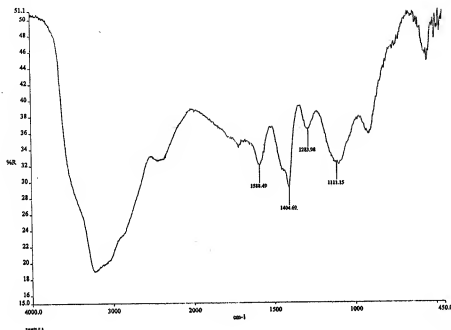
SAMPLE 1 – Present Invention



SAMPLE 2 – '090 Reference



SAMPLE 3 – '307 Reference



The important and distinct differences between the spectra are:

1. The alkyl region from 2950 to 2850 cm^{-1} is much more pronounced in Sample 1 – Present invention. This region is hardly noticeable in both ACTAGRO Samples 2 and 3.
2. The absorbance centered around 1590 cm^{-1} is much stronger in Sample 1 – Present invention than in both ACTAGRO Samples 2 and 3. Moreover, this band is much more intense than the absorption at about 1390 cm^{-1} in Sample 1 – Present invention, whereas the reverse is true in Samples 2 and 3. While the two bands 1590 cm^{-1} and 1390 cm^{-1} can be attributed to the symmetric and asymmetric stretching of carboxylate ions, the one at 1590 cm^{-1} may also contain the skeletal stretchings of alkyl groups and those of different types of ketones. Based on the DRIFT spectra, Sample 1 – Present invention contains both more carboxylic and carbonylic compounds than Samples 2 and 3. This is in agreement with the larger alkyl content of Sample 1 – Present invention when compared to Samples 2 and 3, as it is inferred by the 2950 to 2850 cm^{-1} peaks.

4. The region between 1193 and 1100 cm^{-1} that can be attributed to phenols and alcohols is more distinct in Sample 1, whereas the same region, though intense, seems to be made of more overlapping absorptions in Samples 2 and 3.

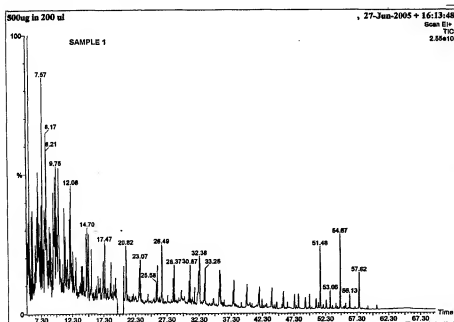
Pyrolysis-Gas Chromatography-Mass Spectrometry (off-line)

Pyrolysis under inert atmosphere is an diagnostic analytical method for the molecular evaluation of humic matter. The pyrolysates are conveyed into a Gas Chromatography-Mass Spectrometry (GC-MS) apparatus in order to obtain a pyrogram that is specific to each single humic material. The humic analysis by pyrolysis is enhanced by a prior treatment of the sample with tetramethylammonium hydroxide (TMAH) in order to methylate the acidic functions of humic molecules and increase their transfer into the gaseous phase.

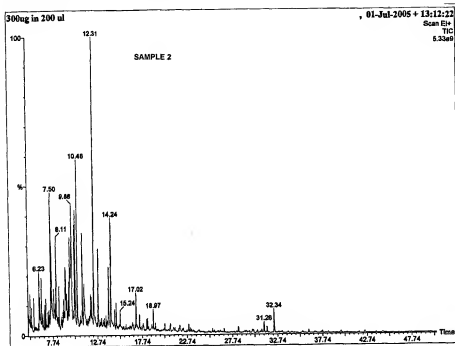
The three selected samples were subjected to an off-line pyrolysis. Briefly, the sample under analysis (from 100 to 200 mg) is treated with 1 mL of TMAH and placed in a glass tube positioned into a circular oven at 400°C. A flow of Helium from one end of the glass tube conveys the pyrolysates into a three consecutive traps of chloroform cooled by ice where the pyrolysates are collected. The chloroform solutions are dried by rotoevaporation and the residue dissolved in n-hexane or dichloromethane prior to the injection into the GC-MS apparatus.

The following are the pyrograms obtained for the selected three samples:

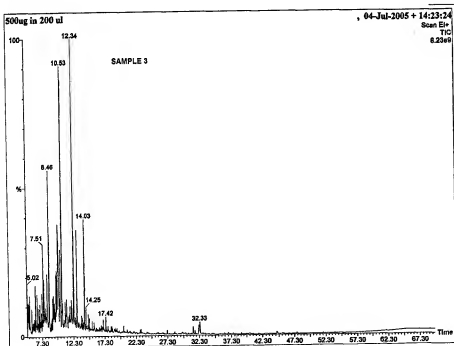
SAMPLE 1 – Present Invention



SAMPLE 2 – 090 Reference



SAMPLE 3 – '307 Reference



The programs show that the humic extract obtained with the present invention (Sample 1) is much richer in pyrolysate compounds than the pyrograms resulting from the analysis of Sample 2 – '090 Reference (ACTAGRO) and Sample 3 – '307 Reference (ACTAGRO). Sample 1 – Present invention resembles the pyrogram of humic extract reported in the literature, while both Samples 2 and 3 reveal a much lower number of pyrolysate peaks.

The experimental results outlined above demonstrate that the process of the presently claimed invention is unexpectedly capable of extracting from leonardite ore a different material than that present in the commercial products (ACTAGRO) that purport to be obtained by extraction methods reported in the '090 and '307 references.

The action of gluconic acid at low pH (below 2.5) can produce a humic extract that is different than that obtained by the use of gluconates or gluconic acid plus gluconates at higher pHs (both cited references).

The different mechanism of the present invention acts effectively on the leonardite ore by producing a surprising extract that is richer in carbon, in carboxyl and carbonyl compounds, and yields a pyrogram that is not only richer in pyrolysates (thereby suggesting a larger and more selective extraction efficiency) but also in compounds similar to those obtained in literature by similar pyrolytic techniques.

In view of the above arguments and amendments, Applicants believe the claims are novel and nonobvious and withdrawal of this rejection is respectfully requested.

Summary

In view of the above amendments and remarks, Applicants respectfully request a Notice of Allowance. If the Examiner believes a telephone conference would advance the prosecution of this application, the Examiner is invited to telephone the undersigned at the below-listed telephone number.

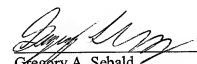
USSN 10/560,081
Reply to Office Action dated 10/14/2008

Please consider this a PETITION FOR EXTENSION OF TIME for a sufficient number of months to enter these papers or any future reply, if appropriate. Please charge any additional fees or credit overpayment to Deposit Account No. 13-2725.

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4/14/09



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